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- (54) **FILTRE ACTIF COMPOSE DE MATERIAU COMPOSITE A GRILLAGE AGGLOMERANT ET PROCEDE DE SEPARATION DE MATIERE ORGANIQUE HYDROPHOBE DU SOL ET DE FLUIDES AQUEUX EN UTILISANT DES TENSIONS D'INTERFACE CONTROLEES ET LA DIMENSION DES PORES**
- (54) **AN ACTIVE FILTER OF COMPOSITE SINTERIZED MATERIAL AND PROCESS FOR SEPARATION OF HYDROPHOBIC ORGANIC MATERIAL FROM SOIL AND AQUEOUS FLUIDS USING CONTROLLED INTERFACE TENSIONS AND PORE SIZES**

(57)

Multi-part polymer composite fused together through a sinterized structure producing a reusable selective filter capable of separating hydrophobic organic materials from an aqueous solution, or soil with the addition of water. System for removing hydrocarbons from both soil and the top and bottom of water. Utilization's include petrochemical extraction from tar sands, oil recovery from oil spills in bodies of water, the purification of used oil as a first step in recycling, liquid/liquid separation, the creation of a reusable fast-breed thin-layer chromatography plate, the creation of higher volume capillary electrophoresis and the recovery of sinking chlorinated compounds.



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(54) **FILTRE ACTIF COMPOSE DE MATERIAU COMPOSITE A
GRILLAGE AGGLOMERANT ET PROCEDE DE
SEPARATION DE MATIERE ORGANIQUE HYDROPHOBE
DU SOL ET DE FLUIDES AQUEUX EN UTILISANT DES
TENSIONS D'INTERFACE CONTROLEES ET LA
DIMENSION DES PORES**

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MATERIAL AND PROCESS FOR SEPARATION OF
HYDROPHOBIC ORGANIC MATERIAL FROM SOIL AND
AQUEOUS FLUIDS USING CONTROLLED INTERFACE
TENSIONS AND PORE SIZES**

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- 1> Abstract
- 2> Prior Inventions
- 3> Differentiation
- 4> Utilization's
- 5> Process
- 6> Claims

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Assignees: Zihnija Hurem, Koy Hong Lee, Blake Kenwell

1>ABSTRACT

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2> PREVIOUS TECHNOLOGIES OF SEPARATION.

There have been a number of earlier patents using the oleophilic, hydrophobic, or absorbent material such as polypropylene, polyethylene, feedstock, alone or in combination with other materials: Herman E Brinkley, US Pat 5,229,006, uses man-made fibers and cloth made from this fiber to absorb hydrocarbons and later applying squeezing force to squeeze out the hydrocarbon so the material can be reused. Steward A Sipson, US Pat. 5,178,769 uses dried fibrous peat as a filter to absorb the hydrocarbon, another sponging process. William E. Sohl, reissue of US Pat 3,764,527, uses another oleophilic fibrous web whose interstitial spaces retain the hydrocarbons. Gerard DeBeukelaer et al, US Pat. 4,279,757, uses a polyurethane foam to absorb the hydrocarbons like a sponge. Walter C Babcock, US Pat. 5,354,469, uses plasma polymer membranes in selective and permeable layers to absorb the hydrocarbons. Stelio Codiglia, US Pat. 5,304,311, uses an ethylene/ alpha- olefin copolymer to absorb hydrocarbon and forms a jelly mass. Richard C Fuisz, US Pat 5,268,110, uses an oil absorbing matrix made from a feedstock to create an oil hog to absorb the oil. All of the above use some form of absorption and sponging action. They are not continuous processes and the absorption substrates have to be processed to be reused or discarded. A further shortcoming of this "sponging" process is that the materials do not pass through the filter and cannot be easily collected.

3> DIFFERENTIATION

This invention is different in that it is a continuous, active-filtering process. Unlike the other patents, this invention permits a flow of the filtrate directly from the solution. This flow works through hydrostatic pressure, and can be assisted with gravity, and or various sizes of pumps. Its utility is not only derived from the absorption qualities of the compound, it is this property in combination with its structural ability to maintain a useful shape capable of permitting a flow of filtrate over its surface for containment. In the same manner that a pipe is useful for directing the flow of liquids, similarly this invention filters hydrophobic organic compounds and is then able to direct them into a container. Pipes can be created out of this material which then has the combined ability to pull in the hydrophobic compounds, and direct them where they are required. With its ability to draw the hydrophobic organic compounds through it, the ZI Filter is able to filter out water and consequently rids the water of the hydrophobic organic compounds. Alternatively the hydrophobic organic compound is purified of any water contaminating it. In application, this permits the pumping of oil from the sea, it permits the pumping of hydrocarbons from soil, with the addition of water, and it permits the pumping of sinking chlorinated solvents such as trichloroethylene from the bottom of any body of water. It also has the effect of being able to remove water from fuel. As a filtered pump it is not limited by the capacity of the absorption qualities of the materials, as are the other patents.

4> UTILITY

This material, ZI Filter, is stable both in its chemical makeup, and in its ability to retain its structure, and consequently is useful both from the point of its properties in relation to hydrophobic organic compounds, and the functional abilities permitted by the solid shapes it can be formed into.

This application relates to an invention whereby hydrophobic organic materials are drawn out of an aqueous solution. Through the application of a variety of shapes, the hydrophobic organic materials can be pumped out of the soil or water. In situations such as oil spills in large bodies of water such as oceans and lakes, down to small containers, water treatment systems, and oil recovery systems, hydrophobic organic products are drawn through a filter and can be recovered. Used motor oil can be purified for recycling purposes. Oil spills can be recovered quickly without danger to the environment and with recovery of the oil for use. Sinking chlorinated solvents like Trichloroethylene, and others, can be safely retrieved from our waterways. Spills in soil can be extracted. Systems of recovery can be implemented under buildings and structures with high risks of spills to prevent environmental contamination, and permit the recovery of compounds spilled.

The present invention is the creation of a new material with specific characteristics capable of separating hydrophobic organic materials from water, including hydrocarbons, and sinking chlorinated solvents. The polymer compound of Teflon™

Polyethylene and Polypropylene is one which can be molded into any shape required and is reusable. By altering the granular sizes, the proportion of the component mix, the temperature and pressure at which the compound is created, and the addition of modifiers, the active filtering properties of the ZI Filter is controlled thereby making it possible to separate various components of the mixture. This invention is thereby capable of being utilized for fast-breed thin-layer chromatography plate and larger volume capillary electrophoresis. It is useful in filtering out unwanted hydrophobic organic compounds in water for release into the sewage system or treatment processes, or alternatively for treating water for drinking purposes.

This invention provides the high speed recovery of oil spilled in lakes rivers or oceans, the remediation of soil, and the recovery of oil in tar sands.

The filter system using this material provides the active filtration of the hydrophobic organic materials out of the mixture, and to recover them in a usable format. Through the use of this material in this fashion, the oil spilled is not only stopped from spreading, which is effectively achieved though surrounding any spill using this material, but the oil spilled is quickly recovered in a usable format.

This invention is also useful in preventing water from getting into gas tanks or, once there to filter out unwanted materials such as water or dirt or other particles from getting into the combustion chamber of an internal combustion engine. This can be done by putting a Zi filter at the point the gas is added and/or in the fuel line going to the engine.

Reusable Fast-breed thin-layer chromatography plate.

Current state of technology in Thin-layer Chromatography utilizes a thin layer of colloid particles applied on glass plate as a one-time use system. The active colloidal layer is very fragile and sensitive to mechanical damages. The actual quantity of analyte is very small and very often not sufficient for further instrumental analysis.

With this invention, using the ZI Filter, we created a three-dimensional separation plate that is self-holding (without the glass bottom support). As a result, the amount of analyte is significantly larger and easier to collect from the chromatography plate. Beyond the utility of analysis only, this invention opens up thin-layer chromatography into use for preparation and purification in chemical production. This invention is also reusable in unlimited number of repeat applications.

Capillary Electrophoresis

Current state of technology uses a single capillary tube through which an analyte is being drawn by an electric field. The amount of analyte is very small and only enough for detection.

This invention using the same plate as described above in thin-layer chromatographic use except for the addition of electropotential force. The microstructure of the ZI Filter acts in effect like many capillary tubes together, with the result that we achieve the same rate of separation but with many orders of magnitude increase in product of analyte. Again this provides utility as a production device beyond analysis.

Liquid/Liquid Extraction.

Current state of technology in liquid/liquid extraction utilizes a separation funnel to separate one liquid analyte from another. The cutoff point is difficult to achieve. With this invention, we have continuous extraction without human attendance. The preferential method for achieving this is to introduce a ZI Filter in the shape of an inverted boot in the bottom of a container. The filtrate is then collected. This approach is applied to collect hydrophobic organic compounds which are heavier than the solution. In cases where the filtrate to be collected is lighter or throughout the mixture the boot need only be placed completely through the mixture.

Oil Recovery; Oil Spills.

In this application, the current state of technology for oil spills is to try and contain the oil slick with a ring of floatation devices, a ring of buoy-like structure. The oil slick has a high chance of spreading once turbulence in the body of water occurs, as in the high seas, and massive disaster occurs. Utilizing this invention, the ZI Filter is made in a modular series of vertical floatation structures that has interlock devices on each side for quick and flexible connections, to create a ring of containment plus active recovery. The inner wall is made of the ZI Filter material with an outside flat chamber that serves as the collection well. One or more tubes are connected into the collection well portion that then runs into other pipes that finally ends into a pump or series of pumps. The preferred example for an oil spill is that within hours of a disaster, a system of the ZI Filter Recovery Panels is air-lifted to the site to be connected into a ring surrounding the oil slick, another oil tanker is dispatched to the scene to pump out the slick. The ZI Filter in this case is constructed with high speed of extraction, which means some water is extracted together with the oil spill. But the net effect is very little oil will be left on the surface for environmental damage. Providing the recovery tanker with a second ZI Filter will purify the oil.

5> THE MANUFACTURING PROCESS FOR THIS FILTER (*called the ZI Filter*)

The polymer compound is made from sinterized Polyethylene and Polypropylene with Polytetrafluorethene (Teflon™) as an interface tension modifier. Depending on the selectivity required further modifiers can be added to the surface as required. These modifiers include compounds with molecular structure of C18 and higher. The Teflon coating ranges in size from 100 to 1000 microns depending on selectivity required. The compound is fused together just under melting temperature.

The final properties of this filter depends on the control of the following factors during sinterization:

1. **Composition.** The blend of the polymers and other materials determines the structural matrix for the ZI Filter and its selectivity for hydrophobic organic compounds. In the first preferred rendering of this process, we use Polyethylene, Polypropylene and Teflon™. The Polyethylene and Polypropylene together act as the matrix, Teflon as the major modifier.

2. **Particle size of the component materials.** The size of the particles influence the size of the pores in the ZI Filter and consequently its selectivity and rate of flow.
3. **Temperature.** In the process, we heat up the components to just below the melting point of the component with the lowest melting point. At this temperature, the particles are pushed against each other in a sinter and not melted into one.
4. **Pressure.** Pressure is applied to the process and this determines the pore size. The range of pressure varies between -10 psi to +100 psi.
5. **Modifier(s).** Certain materials can be added to line the inner surface of the pores and act as selective affinity promoters for the materials we want it to pull through the filter. The modifier is incorporated on to the filter matrix either by exposing the filter to a liquid solution, melted state, or a vaporized state of the modifier. Effective modifiers include molecular structures having a C18 and higher. One example of a modifier is paraffin.
6. **The Mold** used determines the shape of the filter. The preferential material for the mold is steel, alternatively it may be made of other materials like glass or metallic composites to produce an inner smooth surface.

The components are granulated, mixed and put in a mold under pressure and heated to just below the melting point of the lowest melting point component and the ZI Filter is formed.

EXAMPLE OF COMPOSITION

The first preferred composition of the ZI Filter is composed of 40% - 55% polyethylene, 40-55% Polypropylene and 1-10% Teflon. The pressure being applied is between -10psi to +10psi. The variations provide for differences in the structural rigidity and speed of flow through the ZI Filter, dependent upon the purpose for which the ZI Filter is being put.

STRUCTURAL AND SHAPE APPLICATIONS.

Without limiting the scope of this invention, there are specific shapes that are useful as illustrations of the ZI Filter

Closed-End Tube (*The Boot*)

A pipe with a closed end made up of ZI Filter. As a result of its closed end the filtrate will pool inside the boot, and can then be removed through the flow of gravity or with the assistance of a pump. The boot is preferentially useful to introduce into the center of the mixture to be separated.

Coaxial Pipe.

In this application of the process, a coaxial pipe system is made consisting of two or more pipes one inside the other step-wise in selectivity properties.

Buoy Style Oil Recovery System

Sausage like buoys are created out of the ZI Filter for circling oil spills. The buoys are attached to each other by flexible heavy rubber tubes in this manner the oil filtrated through to the center of the filter can be pumped through the line and out of the water.

Pipe Root Structure

A gathering structure is assembled from tubular ZI Filters, for short and long term recovery of hydrophobic organic compounds. One example would be petroleum extraction from tar sands. Alternatively this can be placed in waterways, or sewers to collect contaminants as a permanent structure.

Panel Shapes (The Wall)

Used in the accumulation of oil after a spill in a body of water, or placed in a trench to collect hydrophobic organic compounds, such as oil, or gas. The wall is preferentially used to be introduced at the edge of the mixture to be separated, and to prevent further outflow of contaminants. **Double Wall:** The double wall has a two absoorbant pannels back to back with a collection well in the center: used for the accumulation of hydrophobic organic compounds when placed in the middle of the mixture to be separated. Permits filtration on both sides of the wall.

Leachate Pads

In manufacturing a flat horizontal sheet of the ZI Filter, a network of small collection pipes is laid into the structure of the ZI Filter. This network of collection pipes is made of a non-filter material, such as polypropylene, and serves to collect the run-off from the filter to be collected at one point. The bottom of the ZI Filter is a sheet of impermeable material. The whole is called the "Leachate Pad". This Leachate Pad has the utility of being placed at the bottom of a quantity of material, such as soil, that has been contaminated with organic compounds, such as petrochemicals, and as time goes by, naturally the leachate that runs down to the bottom will contain a mixture of water and oils. The Leachate Pad therefore separates the filtrate, mainly petrochemicals and collects it for further processing, thus also preventing the leachate from spreading deeper into ground water tables or adjacent environment. Other examples of the utility of this Leachate Pad are incorporation into the base of building structures that are used for potentially hazardous spillage activities such as chemical manufacturing. it is also useful as a preventative measure under tanks containing hydrophobic organic compounds.

6> Claims**I Claim:**

1. A new material of sinterized polymers, it has specific useful properties for the separation of hydrophobic organic compounds from aqueous solutions. it also is able to do liquid/liquid extractions with the addition of modifiers. These functions arise from the principles of micropore sinterization, interface tension, and structural soundness providing the foundation for recovering compounds in an active filtering

6> Claims

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process, whereby it is possible to pump these out of the solution in a continuous process, in a reusable format.

2. Process of Manufacture of the new material in claim 1, known as ZI Filter.

The process is the combination of polyethylene, polypropylene and Teflon in a sinterized format, where the components are granulated, mixed and put in a mold under pressure and heated to the just below the lowest melting point component and the ZI Filter is formed.

3. The structure and shape of claim 1 in the shape of flat panels (known as the Wall). (Diagram 7)

4. The structure and shape of claim 1 in the shape of flat panels back to back, (Known as the Double Wall). (Diagram 9)

5. The structure and shape of claim 1 in the shape of a pipe with a closed end (known as the boot). (Diagrams 1 & 2)

6. The structure and shape of claim 1 in the shape of a horizontal panel with a hydrophobic organic compound impermeable liner on the bottom, and impregnated with a network of small collection pipes (known as the Leachate Pad).

7. The structure and shape of claim 1 in the shape of one pipe in another with varying ZI Filter properties, (known as Coaxial Pipe). (Diagram 4)

8. The structure and shape of claim 1 in an assembly of root like pipes merging into a collection retainer (known as a Pipe Root Structure). (Diagram 6)

9. Oil Spill Recovery system using claim 3 in a large format, the ZI Filter is made in a modular series of vertical floatation structures that have interlock devices on each side for quick and flexible connections to create a ring of containment plus active recovery. The inner wall is made of the ZI Filter material with an outer wall outside flat chamber that serves as the collection well. One or more tubes are connected into the collection well that then run into other pipes ending in a pump system. This pump system flows through a second ZI Filter to remove any water still in the oil, and the filtrate is then put in the hold of the tanker for reuse. (Diagram 8)

10. A reusable Fast-Breed Thin-Layer Chromatography Plate, using the material in claim 1 in the shape of claim 3, sized according to need. This Chromatography Plate is reusable, and not sensitive to mechanical damage. As a result the amount of analyte is significantly larger and easier to collect than previous thin layer chromatography.

11. Beyond the utility of analysis of claim 10, this material of claim 1 elevates thin layer chromatography into a process for preparation and purification in chemical production.

12. The use of ZI Filter, the material of claim 1, in Fast Breed Capillary Electrophoresis Similar in structure to claim 11, with the addition of electropotential force. The micro structure of the ZI Filter acts like many capillary tubes together with the result that we achieve the same rate of separation, but with many orders of magnitude increased in volume of analyte. This provides utility as a production device beyond analysis.

13. The use of ZI Filter, the Material of Claim 1, in the preferred shape of claim 5, the boot, in the process of recovering sinking chlorinated solvents from the bottoms of bodies of water. The boot is weighted and sealed airtight with a connection tube to a pump, drawing the solvents into a container. This is dropped into the bottom of the body of water and the solvent is consequently pumped out. (Diagram 3)

14. The use of ZI Filter, the material of claim 1, in the process of Water Purification, specifically for the removal of hydrophobic organic compounds. This is accomplished in a preferred method through the use of either claim 5, or alternatively 8, depending on the structure already in place for water treatment. The ZI Filter is made small and numerous to provide maximum efficiency in surface area contact.

15. The use of the ZI Filter, the material of claim 1, in the shape of an inverted boot (claim 5), for the purpose of Liquid/Liquid Extraction.

16. The use of ZI Filter the material of claim 1, to be placed in the fuel line of any internal combustion engine, for the use of excluding water from the combustion chamber.

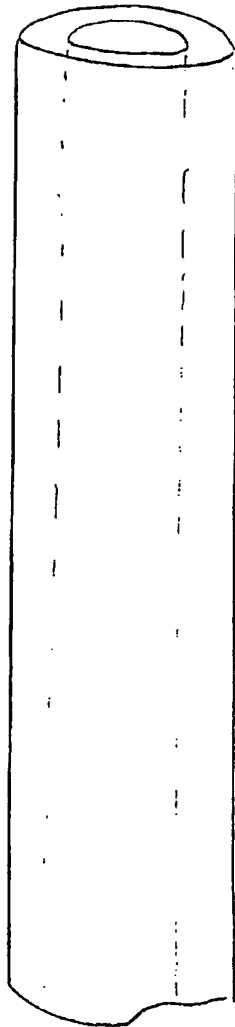
17. The use of ZI Filter, the material of claim 1, in the shape of claim 5, Hydrophobic Organic Compound purification system for the use of removing impurities.

18. The use of ZI Filter, the material of claim 1, in the shape of claim 3 or 5, in the first stage of recycling used oil, removing oxidized oil, and physical impurities, such as dirt and metals, to prepare the oil for refining to have the additives replaced for reuse. Without this stage the oil would not be suitable for processing.

19. The use of ZI Filter, the material in claim 1, for a Petroleum extraction system for tar sands. Specifically the process of embedding the form of claim 8 in the sands and pumping out the resultant petroleum filtrate Alternatively a leachate solution may be applied, where the soil is dug up and deposited on a leachate pad for recovery of petrochemicals.

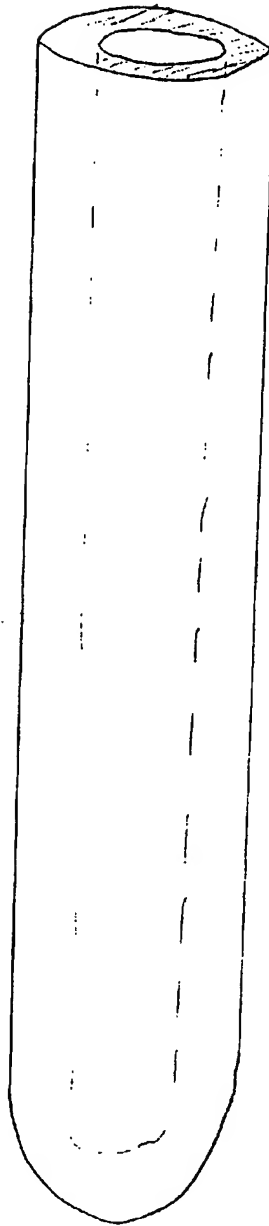
20. The use of ZI Filter, the material in claim 1, for the remediation of water contaminated with hydrophobic organic compounds, using the form of claim 5, the boot.

21. The use of ZI Filter, the material in claim 1, for the remediation of soil contaminated with hydrophobic organic compounds, using the form of claim 3, the wall. The soil is washed with hot water, and the wall is set up in a trench at the lowest elevation, consistent with the geological formation of the land. The contaminants are absorbed by the wall and contained outside of the soil.
22. Method of 2 whereby the ZI Filter is altered to be selective by the alteration of Teflon thickness, between 100 to 1000 microns.
23. Method of 2 whereby the ZI Filter is altered to be selective by the addition of Modifiers from molecular structures containing C18 through to and including C100.
24. Method of 2 whereby the ZI Filter is altered to be selective by the method of imbibing of the C18 through to and including C100 molecular structures specifically through addition in solid.
25. Method of 2 whereby the ZI Filter is altered to be selective by the method of imbibing of the C18 through to and including C100 molecular structures specifically through addition in liquid state.
26. Method of 2 whereby the ZI Filter is altered to be selective by the method of imbibing of the C18 through to and including C100 molecular structures specifically through addition in vapour state.
27. Method of 2 whereby the ZI Filter is altered to be selective by the alteration of pressure, from -10psi up to and including 50,000 psi.
28. Method of 2 whereby the ZI Filter is altered to be selective by the size of the granules of polyethylene from 100 micron to 100,000 microns.
29. Method of 2 whereby the selectivity and structural capability of composition ratio of sinterized polyethylene, Polypropylene and Teflon range between 0- 100%/0-100%/0-30%.
30. The structure and shape of claim 1, in the form of tubular components, joined together with a rubber hose. The rubber hose is clamped from the inside with a bolt and washer and protected with a strap. The outside is also clamped with a strap and bolted into place. A chain of these are formed around oil spills, preferable three, at minimum one. The filtrate is pumped from the center of these buoys as they form a filtering hose for deposit into a second filter to remove any water, and then into the ship for transport and reuse. (Diagram 5)



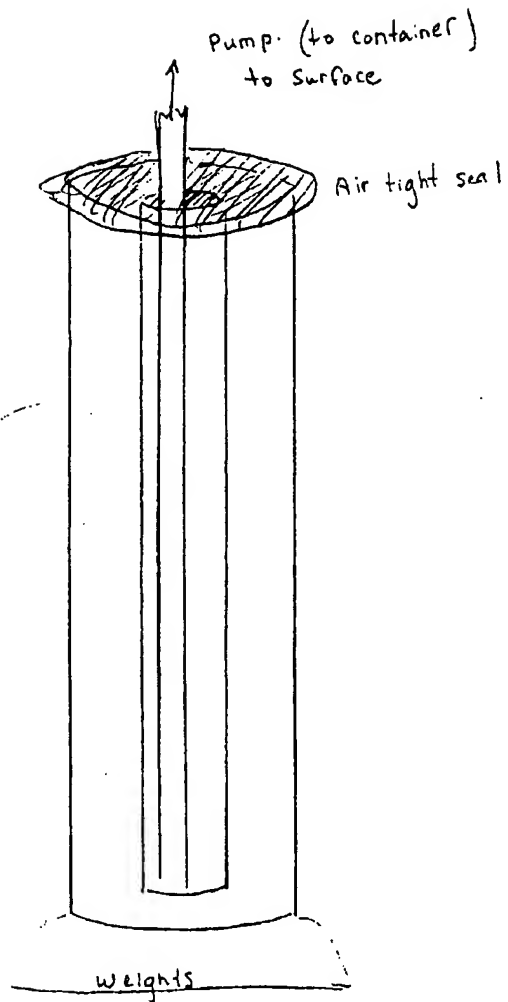
Pipe

Diagram 1

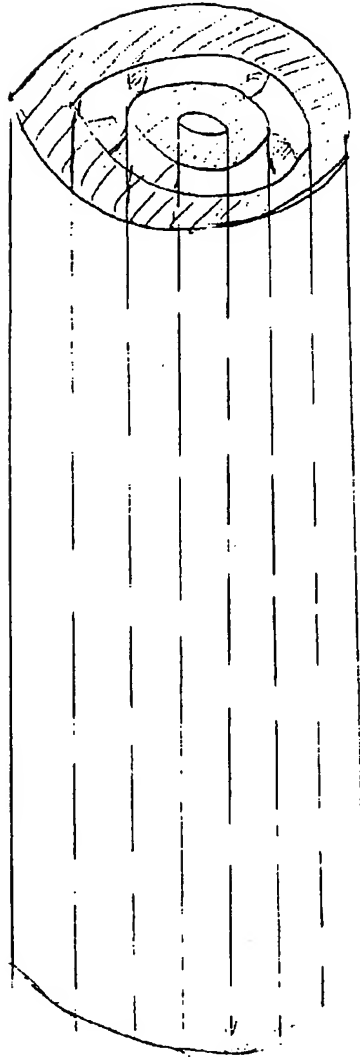


'Boot'

Diagram 2

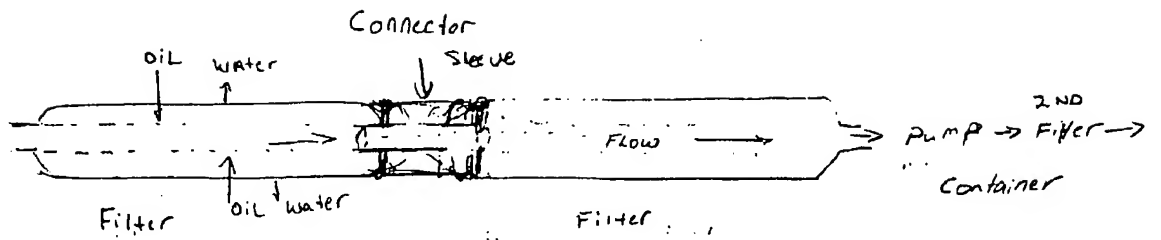


'Boot' with weight
Diagram 3



Coaxial Pipe

Diagram 4



Buoy style oil Recovery system.

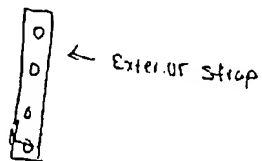
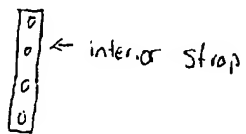
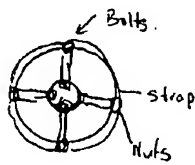
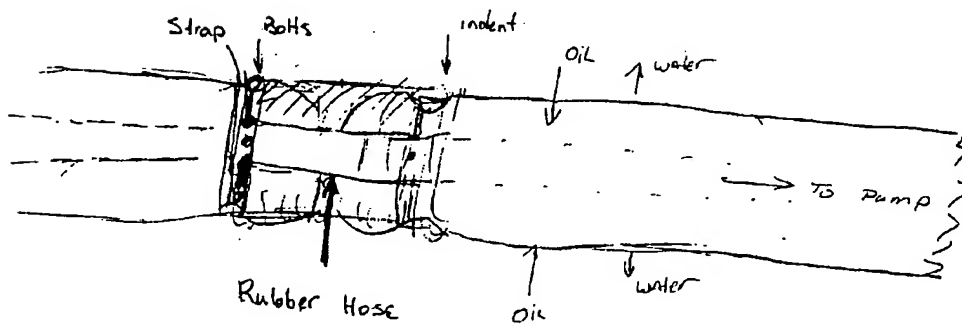
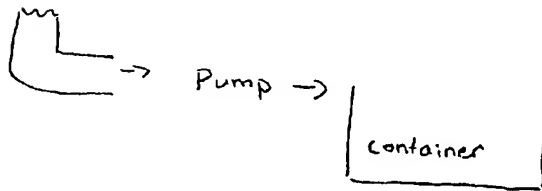
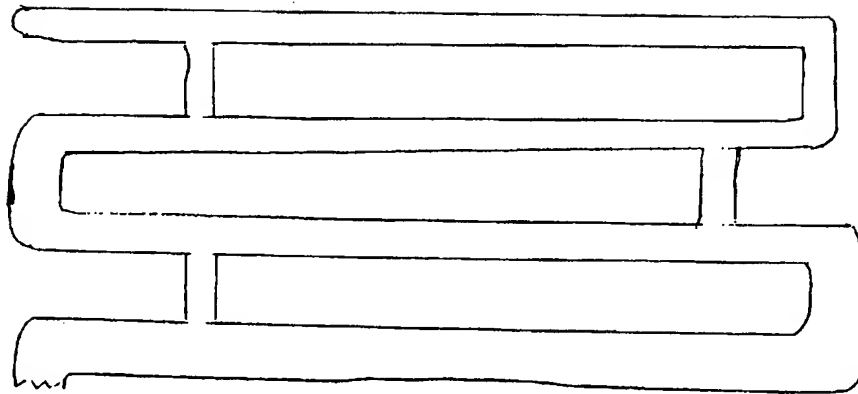
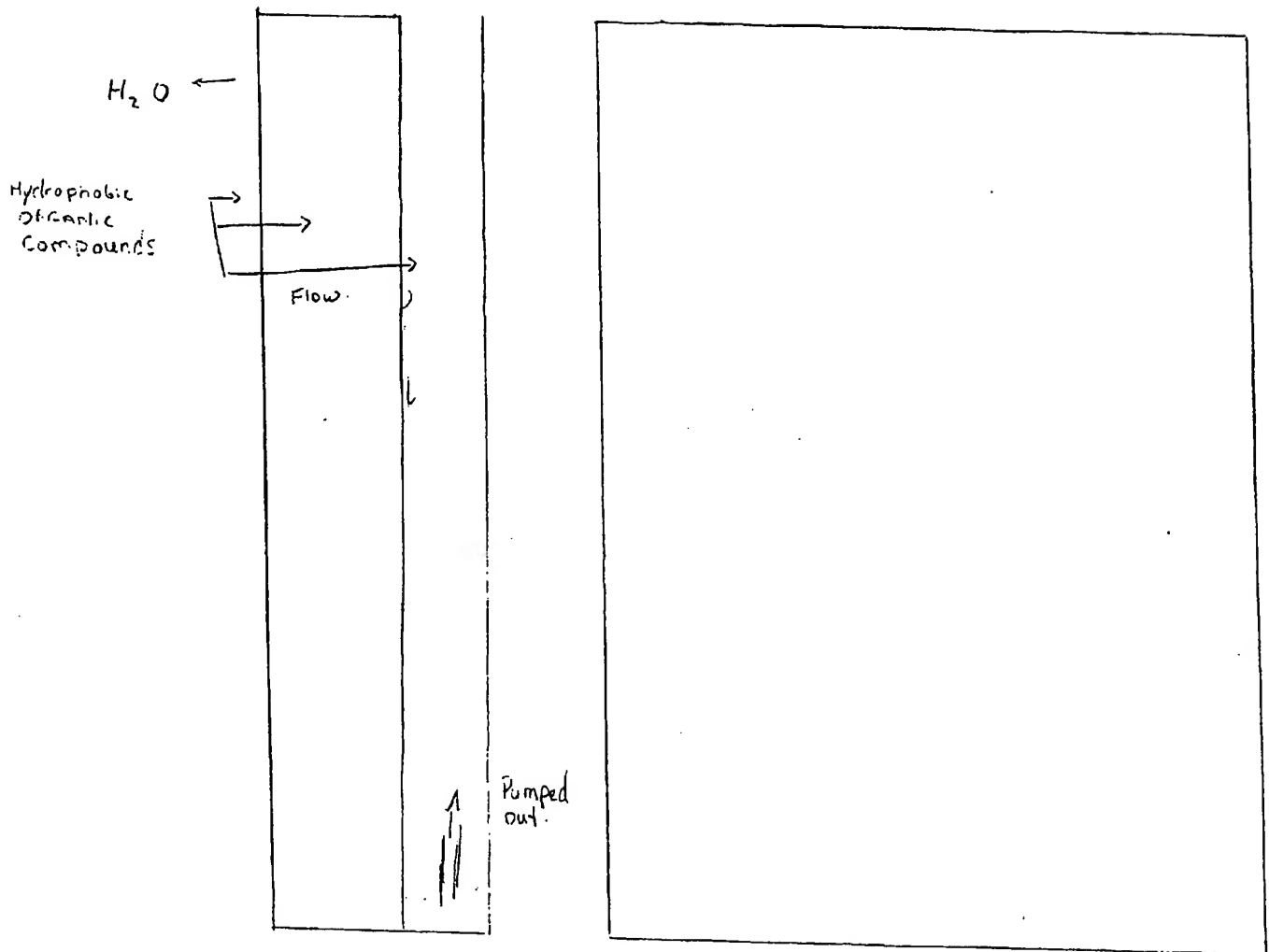


Diagram 5



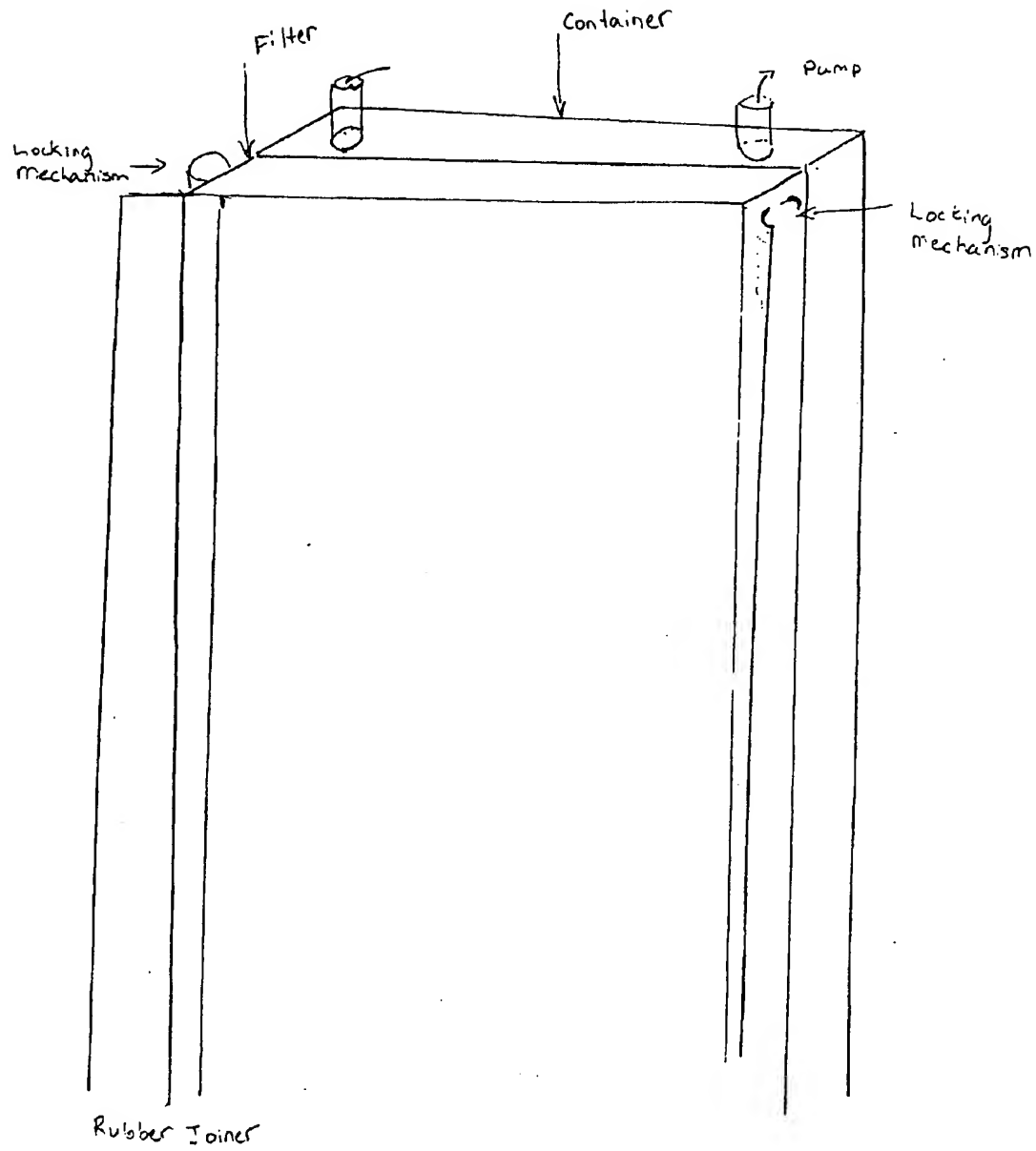
'Root' pipe

Diagram 6



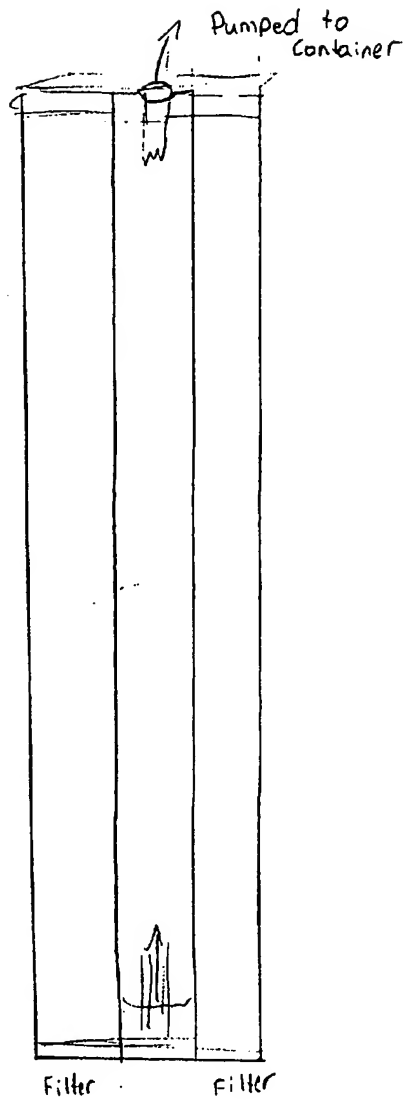
'Wall'

Diagram 7



Oil Recovery Wall

Diagram 8



Double Wall

Diagram 9